

**Claims**

1. A method of performing Nuclear Magnetic Resonance (NMR) spectroscopy on a hyperpolarized sample, which method comprises the steps of:
  - hyperpolarizing a sample using DNP, wherein the NMR active nuclei receive hyperpolarization;
  - performing NMR spectroscopy on the sample with the use of sequences of rf-pulses, wherein the pulse sequences comprises at least two rf-pulses, either on the same nuclei or on different nuclei, and wherein the pulse sequence is adapted for a hyperpolarized sample, thereby producing at least two NMR spectra;
  - analysing at least two of the NMR spectra in order to obtain a characterization of the sample, or to obtain an interim result to be used in the NMR spectroscopy step.
2. The NMR spectroscopy method according to claim 1, wherein the pulse sequence is adapted to the hyperpolarized sample by using a single scan pulse sequence in which one initial 90° pulse is followed by a plurality of spin echo pulses.
3. The NMR spectroscopy method according to claim 1, wherein the pulse sequence is adapted to the hyperpolarized sample by using repeated excitation pulses with small flip angles in order to maintain at least a portion of the hyperpolarization after the initial excitation pulse.
4. The NMR spectroscopy method according to claim 1, wherein the flip angle,  $\alpha$ , of the excitation pulse is varied during the pulse sequence.
5. The NMR spectroscopy method according to claim 1, wherein the flip angle,  $\alpha$ , of the excitation pulse is essentially constant during the pulse sequence.
6. The NMR spectroscopy method according to claim 1, wherein the flip angle,  $\alpha$  is increased in each repetition such that an equal amount of transverse magnetization is created, and such that the last excitation pulse is a 90° pulse.
7. The NMR spectroscopy method according to claim 1, wherein the method is used for multidimensional NMR spectroscopy, which method comprises:
  - a pulse sequence which comprises pulses followed by evolution periods, enabling a

mapping of a multidimensional time-space grid, in which the time parameters are modulated by different coupling constants, said pulse sequence being adapted to take into account the initial hyperpolarized spin state, and

- a subsequent analysis of the multidimensional time domain data, which analysis comprises a transformation of the time domain data to frequency domain data.

8. The NMR spectroscopy method according to claim 8, wherein the multidimensional time space is explored through a trajectory that spans all parts of the time space that provide essential information used in subsequent analysis.

9. The NMR spectroscopy method according to claim 9, wherein the multidimensional time space is two dimensional and the trajectory is present in all quadrants of the two time plane.

10. The NMR spectroscopy method according to claim 1, wherein the spectroscopy step comprises:

- recording a first one-dimensional spectrum (100) for a first nuclear spin species in the sample;

- recording a second one-dimensional spectrum (110) for a second nuclear spin species in the sample;

and step of analyzing comprise the steps of:

- identifying (120) in the first spectra singlet peaks and corresponding doublets;

- correlating (130) peaks or groups of peaks in the two spectras by comparing and identifying the same splitting, whereby determining which first nuclear spin species are scalarly coupled to a specific nuclei of the second nuclear spin species.

11. The NMR spectroscopy method according to claim 1, wherein the spectroscopy step comprises:

- recording a first spectrum (200) for a first (I) nuclei that is to be decoupled;

- performing a decoupling procedure (210) on the first nuclear spin species (I) spins and a simultaneously monitoring of a second nuclear spin species (S) by:

- subjecting the second spin species (S) to a pulse sequence (210:1), which sequence comprises a first 90° pulse which transfers the entire magnetization of the S spins to the transverse plane; and a subsequent train of refocusing pulses (spin echo pulses), during

which train of refocusing pulses a different decoupler frequency is used during each refocusing pulse.

12. The NMR spectroscopy method according to any of claims 1-1, wherein the spectroscopy step comprises:

- recording a first spectrum (200) for a first (I) nuclei; that is to be decoupled;
- performing a decoupling procedure (210) on the first nuclear spin species (I) spins and a simultaneously monitoring of a second nuclear spin species (S) by:
- subjecting the second spin species (S) to a pulse sequence (210:2), which sequence comprises a plurality of small flip angle pulses.

13. The NMR spectroscopy method according to any of claims 1-1, wherein the pulse sequence is a diffusion weighted sequence adapted for a hyperpolarized sample.

14. The NMR spectroscopy method according to any of claims 8-10, wherein the pulse sequence is performed on a hyperpolarized sample which comprises a first nuclear spin species (I) and a second nuclear spin species (S), the pulse sequence comprises the step of:

- (300) starting from the point (0,0), with an  $90^\circ$  pulse on S, observing  $N+1$  points (i,i) up to point (N,N);
- (305) performing a pulse I (i.e.  $180^\circ$  on I), which leads to (N,-N);
- (310) waiting one time unit, leading to (N+1,-N+1);
- (315) performing a pulse IS (a  $180^\circ$  pulse on both I and S), leading to (-N-1,-N+1);
- (320) observing points up to (N-2,N);
- (325) performing a pulse I, leading to (N-2,-N);
- (330) observing points up to (N+1,-N+3).

15. The NMR spectroscopy method according to any of claims 8-10, wherein the pulse sequence is performed on a hyperpolarized sample which comprises a first nuclear spin species (I) and a second nuclear spin species (S), the pulse sequence comprises the step of:

- (300b) starting from the point (0,0), with an  $90^\circ$  pulse on S, observing  $N+1$  points (i,i) up to point (N,N);
- (305b) performing a pulse I (i.e.  $180^\circ$  on I), which leads to (N,-N);
- (310b) waiting one time unit, leading to (N+1,-N+1);

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- (315b) performing a pulse IS (a  $180^\circ$  pulse on both I and S), leading to  $(-N-1, -N+1)$ ;
  - (320b) observing points up to  $(N-2, N)$ ;
  - (340b) performing a pulse S, which reverses both time signs and leads to  $(-N+2, -N)$ ;
  - (345b) observing points up to  $(N, N-2)$ ;
  - (350b) performing a pulse I leading to  $(N, -N+2)$ ;
  - (355b) observing points up to  $(N+1, -N+3)$ .